

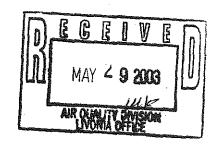
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AIR ENFORCEMENT BRANCH, U.S. EPA, REGION 5

May 27, 2003

Chris Ethridge Environmental Quality Analyst MDEQ, SE Michigan District Headquarters 38980 7 Mile Road Livonia, MI 48152



Re: 2003 RATA for Boiler #10 - Permit No. 184-01A

Dear Mr. Ethridge;

Please find enclosed a copy of the annual RATA for Boiler 10.

The RATA was completed on April 29, 2003. The NOx Relative Accuracy (RA) for Boiler 10 was 8.2%, stated as lb/MMbtu.

If you have any questions please call me at 810-326-2763.

Sincerely,

Daniel Taylor

Environmental Manager

cc:

Tom Gasloli

MDEQ – Air Quality Division 525West Allegan Street – 3rd Floor

Lansing, MI 48909

Daniel Am

Gene Chauffe Don Chutas Wade Richards Test Report for
Compliance Testing of EU BOILER 10
While Firing Natural Gas
at Cargill Salt, Inc.
Permit No. 184-01A

Test Date: April 29, 2003

Prepared For:

Cargill Salt Inc.
916 South Riverside Avenue
St. Clair, Michigan 48079

Project Number 16-030329-00

May 16, 2003

Prepared by: NTH Consultants, Ltd. Todd Wessel (616) 575-1022 Barry L. Broering (616) 575-1011

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EXECUTIVE SUMMARY

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt, Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_X) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only. The testing was conducted with Boiler 10 operating at greater than fifty (50) percent of the steam ouput capacity (150,000 pounds per hour faceplate rating).

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B. The results of all testing can be found in Table 1, located at the end of this report, and is summarized below.

EXECUTIVE SUMMARY TABLE

SUMMARY OF LB/MMBTU RELATIVE ACCURACY RESULTS

SOURCE	TEST PARAMETER	RELATIVE ACCURACY (based upon reference method)	ALLOWABLE LIMITS (based on mean value)
EU BOILER 10	NO _x	8.2 %	20 %



1.0 INTRODUCTION

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt, Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_X) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only.

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B.

NTH personnel Barry Broering and Leona Taylor performed the compliance testing on April 29, 2003. The testing was coordinated through Mr. Dan Taylor of Cargill.

2.0 PROCESS DESCRIPTION

The process boiler identified as EU BOILER 10 is a Nebraska N2S-7-89-ECON model that is designed to combust either NG or No. 2 fuel oil. The primary fuel used in the boiler is NG, with the No. 2 fuel oil serving as a backup fuel source (annual capacity factor for No. 2 oil firing is 10% or 1,176,526 gallons/year). The boiler is used to provide process steam for various plant operations and is rated at a maximum heat input of 184 Mmbtu/hour and a steam output capacity of 150,000 pounds per hour. When firing NG with an approximate heating value of 1,050 Btu/scf, the boiler will consume about 175,238 scf of NG per hour at full load.



3.0 EMISSION RATE SAMPLING AND ANALYTICAL PROCEDURES

The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db.

The following U.S. EPA test methods were used during the emission rate testing:

- Method 3A Determination of Oxygen and Carbon Dioxide Concentrations in Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 6C Determination of Sulfur Dioxide Emissions From Stationary Sources
 (quality control requirements of 6C applied to Methods 3A and 7E);
- Method 7E Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 19 Determination of Sulfur Dioxide Removal Efficiency and Particulate
 Matter, Sulfur Dioxide, and Nitrogen Oxide Emission Rates

3.1 Flue Gas Composition Dry Molecular Weight

The flue gas composition was determined using U.S. EPA Reference Method 3A "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)." The carbon dioxide was used for flue gas composition and pound per million british thermal unit (Lb/MMbtu) emission calculations.

3.2 CONTINUOUS EMISSION MONITORING SYSTEM (CEMS)

For all CEMS sampling, the NTH Reference Monitors (RM) require that the effluent gas sample be conditioned to eliminate any possible interference (i.e., water vapor and/or particulate matter) before being transported and injected into each analyzer. All components of the sampling system which contact the sample were constructed of Type 316 stainless steel or Teflon. The signal output from each monitor was connected to a computerized data acquisition system (DAS). The CO₂ and NO_x sample collection system consisted of a heated probe



with a particulate filter, heated sample line, a moisture removal trap, a secondary particulate filter and a sample pump. The sample was collected from the stack and routed through a distribution manifold for delivery to the analyzers. The configuration of the sampling system allowed for the injection of calibration gases directly to the analyzers or through the sampling system. All monitors in use were calibrated with U.S. EPA Protocol No. 1 calibration gases and operated to insure that zero drift, calibration gas drift, and calibration error met the specified method requirements. Response times were determined for each analyzer in use and found to be acceptable (30 seconds). Twelve test repetitions (each repetition 21 minutes in duration) were performed. A schematic of the CEMS is shown in Figure 2. Copies of all Protocol No. 1 gas standard certifications are in Appendix D.

3.2.1 Carbon Dioxide

A Horiba Model CMA 331A non-dispersive infra-red (NDIR) analyzer was used to measure CO₂ concentrations following the guidelines of U.S. EPA Method 3A, "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from a Stationary Source (Instrumental Analyzer Procedure)", in conjunction with Performance Specification No. 2 of Appendix B, 40 CFR 60. The analyzer was set at 25% instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C. Calibration error results are presented in Appendix A.

The CO₂ measurement principle utilizes a non-dispersive infrared (NDIR) method to measure the absorption of specific wavelengths of infrared radiation (IR) by the gas molecules through the use of the Gas Filter Correlation (GFC) technique. Radiation from an infrared source is chopped and then passed through a gas filter that alternates between a sample gas and N₂ due to the rotation of a filter wheel. The radiation then enters a multiple optical pass sample cell where absorption by the sample gas occurs. The IR radiation exits the sample cell and passes through a narrow band pass interference filter to fall on an IR detector. The sample gas filter produces a reference beam, which cannot be further affected by sample gas in the sample chamber. The N₂ side of the filter wheel is transparent to IR radiation and therefore produces a beam, which can be absorbed by the sample gas. The chopped detector signal is modulated by the alternation between the two gas filters with



amplitude proportional to the concentration of sample gas in the sample chamber. Other gases do not cause modulation of the detector signal since they absorb both the reference and measuring beams equally. Thus, the Gas Filter Correlation System responds solely to the desired sample gas molecules.

3.2.2 Nitrogen Oxides

The nitrogen oxides concentration was determined using U.S. EPA Reference Method 7E "Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure)". A Thermal Environmental Instruments chemiluminescence analyzer was set at 100 ppm instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C.

The analyzer converts the NO_X to NO and then measures the light emitted by the NO with ozone gas. The sample gas is drawn into the analyzer by a vacuum pump, which partially evacuates the reaction chamber. The sample flows through a NO_2 to NO converter for NO_X analysis or may pass through the converter for NO analysis. The sample then flows through a temperature controlled critical orifice into the partially evacuated reaction chamber. Ambient air is drawn into the analyzer as a source for the generation of ozone. The air flows through a desiccant cartridge for drying, then through an ozone generator converting some of the oxygen to ozone. The ozonated air then flows through a temperature controlled critical orifice into the reaction chamber. The sample gas and the ozonated air are mixed in the reaction chamber, where the following reaction takes place:

$$NO + O_3 \rightarrow NO_2 + O_2 + hv$$

The intensity of the chemiluminescence is proportional to the concentration of NO in the reaction chamber. The light emitted by this chemiluminescent reaction shines through a window in the chamber onto a photomultiplier tube (PMT). A spinning chopper wheel between the reaction chamber and the PMT allows the dark PMT output to be compared electronically with the chemically generated chemiluminescent PMT output. The signal is processed electronically for recording the NO concentration (or NO_X if the converter is used).



A nitrogen dioxide to nitrogen oxide conversion efficiency test was performed on the NTH NO_X analyzer on April 22, 2002. The results of the NO_X converter test can be found in Appendix D.

3.2.3 Data Acquisition System

Information and data collected with the STRATA data acquisition system (DAS) was used for subsequent data reduction and reporting using a Microsoft Excel spreadsheet program. In addition to providing an instantaneous display of analyzer responses, the DAS compiles the analyzer data collected, and documents analyzer calibrations for download to a floppy disc. The DAS logs the average concentration every 10 seconds, or 6 times a minute.

4.0 RELATIVE ACCURACY TEST AUDIT PROCEDURES

The RATA was conducted to meet the requirements of 40 CFR Part 60, Appendix B, Performance Specification 2 and with the sampling and calibration procedures specified in 40 CFR 60, Appendix A, U.S. EPA Methods 3A, 6C, 7E, and 19. All of the RATA testing was conducted while the boiler was operating at more than 50% capacity. As specified in P.S. 2, Section 13.2, the relative accuracy of the CEMS can be calculated on the RM (average emissions are greater than 50% of the emission standard) or on the applicable emission standard (average emissions during test are less than 50% of the emission standard). The Relative Accuracy for this test report is calculated on the RM method.

4.1 Continuous Emissions Monitoring System (Cargill)

The extractive continuous emissions monitoring systems (CEMS) at the Cargill facility is in operation to monitor emissions from Boiler 10 and is installed to comply with Permit 184-01A. The CEM system includes a data acquisition system to provide data averaging. Graphs with averages for each applicable test period are contained in Appendix B.



4.2 Reference Monitoring System (NTH)

The stack gas was extracted from the stack continuously through a heated stainless steel probe. Measurements were conducted at a three-point traverse (approximately 16.7%, 50% and 83.3% of the sample stream diameter). Twelve 21-minute test runs were conducted, and the best nine runs were used to calculate the relative accuracy (RA). A diagram of the reference monitoring system is illustrated in Figure 2.

Data collected from the NO_X, and CO₂ analyzers were averaged for each test run. NO_X concentrations are reported in parts per million (ppm), converted to an emission rate and reported as (lbs/MMBtu), using equation 19-6 of U.S. EPA Method 19 of Appendix A, 40 CFR 60. CO₂ concentrations are reported in percent (%).

5.0 DATA REDUCTION AND CALCULATIONS

The exhaust gas flow rate and other exhaust gas characteristics calculations were performed by importing raw test data into standardized Microsoft Windows $Excel^{TM}$ worksheets. Once in the worksheet, average values were determined and the data was manipulated to return the desired information. The following calculations and procedures detail the determination of CO2, and NO_X emission rates.

The raw concentrations drawn from the stack effluent by the NTH CEMS analyzers was corrected for zero and upscale sampling system bias checks. The following formula was used to determine the corrected CO_2 and NO_X concentrations.

$$C_{gas} = \left(\overline{C} - C_o\right) \times \frac{C_{ma}}{C_m - C_o} \qquad Eq. 6C - I$$

Where:

 C_{gas} = Effluent gas concentration, dry basis, ppm.

 \overline{C} = Average gas concentration indicated by gas analyzer, dry basis, ppm.

 $C_{\rm m}$ = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.

C_{ma} = Actual concentration of the upscale calibration gas, ppm.



After correcting the concentration values, Equation 19-6 found in U.S. EPA Method 19 was used to determine the emission rate in terms of pounds per million Btu heat input:

$$E = C_{\text{(stack)}} \times 1.194 \times 10^{-7} \times F_c \times \left(\frac{100}{\% \text{CO}_{2d}}\right)$$
 Eq.19 - 6

Where:

E = Mass emission rate of pollutant, $lb/10^6$ Btu.

 $C_{\text{(stack)}}$ = Concentration in the stack, ppm by volume, dry basis.

 F_c = Fuel Factor for natural gas. (Table 19-2: 1,040 dscf/10⁶ Btu)

%CO_{2d} = Measured CO₂ concentration, dry basis, percent.

 1.194×10^{-7} = Conversion factor, (ppm NO_X to lb/scf NO_X)

6.0 RESULTS

The best nine out of twelve runs were used to calculate relative accuracy. 40 CFR Part 60, Appendix B, Performance Specification No. 2 states that a maximum of three sets of the test results may be rejected, so long as the total number of test results used is greater than or equal to nine. Test Run 1 was rejected, due to the recalibration of the Cargill NO_X analyzer by the CEMS vendor (Monitoring Solutions).

The RATA results for the NO_X analyzer indicates that the unit is within the allowable limits of 40 CFR, Part 60, Performance Specifications (PS) 2, which state that Relative Accuracy limits are 20 percent of the mean value of the Reference Method (RM) test data. The NO_X Relative Accuracy (RA) for Boiler 10 was 8.2 percent, stated as Lb/MMbtu based on the RM (Table 1).

The following information is appended:

- A Boiler 10 Reference Monitor (NTH) RATA Test Data
- B Boiler 10 CEM System (Cargill) RATA Test Data
- C NTH Field Data
- D QA/QC data

FIGURES &
TABLES &
APPENDICES
NOT COPIED

TABLE



July 9, 2004

Chris Ethridge Environmental Quality Analyst MDEQ, SE Michigan District Headquarters 38980 7 Mile Road Livonia, MI 48152

RECEIVED

MAR 2 2 2006

AIR ENFORCEMENT BRANCH,

U.S. EPA, REGION 5

Re: 2004 RATA for Boiler #10 - Permit No. 184-01A

Dear Mr. Ethridge;

Please find enclosed a copy of the annual RATA for Boiler 10.

The RATA was completed on May 11, 2004. The NOx Relative Accuracy (RA) for Boiler 10 was 3.8%, stated as lb/MMbtu.

If you have any questions please call me at 810-326-2841.

Sincerely,

Wade Richards

Powerhouse Superintendent

cc:

Tom Gasloli

MDEQ – Air Quality Division 525West Allegan Street – 3rd Floor

Lansing, MI 48909

Don Chutas Dan Taylor JUL 1 2 2004
AIR QUALITY DIV.



Test Report for Compliance Relative Accuracy Testing Of EU BOILER 10 at Cargill Salt, Inc. Permit No. 184-01A

Test Date: May 11, 2004

Prepared For:

Cargill Salt Inc.
916 South Riverside Avenue
St. Clair, Michigan 48079

Project Number 16-040298-00

June 15, 2004

Prepared by: NTH Consultants, Ltd. Todd Wessel (616) 575-1022 Barry L. Broering (616) 575-1011



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EXECUTIVE SUMMARY

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_x) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only. The testing was conducted with Boiler 10 operating at greater than fifty percent (50%) of the steam ouput capacity (150,000 pounds per hour faceplate rating).

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B. The results of all testing can be found in Table 1, located at the end of this report, and is summarized below.

EXECUTIVE SUMMARY TABLE

SUMMARY OF LB/MMBTU RELATIVE ACCURACY RESULTS

SOURCE	TEST PARAMETER	RELATIVE ACCURACY (based upon reference method)	ALLOWABLE LIMITS (based on mean value)
EU BOILER 10	NO _x	3.8%	20 %



1.0 INTRODUCTION

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_x) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only.

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B.

NTH personnel Barry Broering and Leona Taylor performed the compliance testing on May 11, 2004. The testing was coordinated through Mr. Dan Taylor of Cargill. Mr. Thomas Gasloli of the MDEQ witnessed the testing.

2.0 PROCESS DESCRIPTION

The process boiler identified as EU BOILER 10 is a Nebraska N2S-7-89-ECON model that is designed to combust either NG or No. 2 fuel oil. The primary fuel used in the boiler is NG, with the No. 2 fuel oil serving as a backup fuel source (annual capacity factor for No. 2 oil firing is 10% or 1,176,526 gallons/year). The boiler is used to provide process steam for various plant operations and is rated at a maximum heat input of 184 Mmbtu/hour and a steam output capacity of 150,000 pounds per hour. When firing NG with an approximate heating value of 1,050 Btu/scf, the boiler will consume about 175,238 scf of NG per hour at full load.



3.0 EMISSION RATE SAMPLING AND ANALYTICAL PROCEDURES

The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db.

The following U.S. EPA test methods were used during the emission rate testing:

- Method 3A Determination of Oxygen and Carbon Dioxide Concentrations in Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 6C Determination of Sulfur Dioxide Emissions From Stationary Sources (quality control requirements of 6C applied to Methods 3A and 7E);
- Method 7E Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 19 Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide, and Nitrogen Oxide Emission Rates

3.1 FLUE GAS COMPOSITION DRY MOLECULAR WEIGHT

The flue gas composition was determined using U.S. EPA Reference Method 3A "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)." The carbon dioxide was used for flue gas composition and pound per million british thermal unit (Lb/MMbtu) emission calculations.

3.2 CONTINUOUS EMISSION MONITORING SYSTEM (CEMS)

For all CEMS sampling, the NTH Reference Monitors (RM) require that the effluent gas sample be conditioned to eliminate any possible interference (i.e., water vapor and/or particulate matter) before being transported and injected into each analyzer. All components of the sampling system which contact the sample were constructed of Type 316 stainless steel or Teflon. The signal output from each monitor was connected to a computerized data acquisition system (DAS). The CO₂ and NO_x sample collection system consisted of a heated probe with a particulate filter, heated sample line, a



moisture removal trap, a secondary particulate filter and a sample pump. The sample was collected from the stack and routed through a distribution manifold for delivery to the analyzers. The configuration of the sampling system allowed for the injection of calibration gases directly to the analyzers or through the sampling system. All monitors in use were calibrated with U.S. EPA Protocol No. 1 calibration gases and operated to insure that zero drift, calibration gas drift, and calibration error met the specified method requirements. Response times were determined for each analyzer in use and found to be acceptable (30 seconds). Twelve test repetitions (each repetition 21 minutes in duration) were performed. A schematic of the CEMS is shown in Figure 2. Copies of all Protocol No. 1 gas standard certifications are in Appendix D.

3.2.1 Carbon Dioxide

A Horiba Model CMA 331A non-dispersive infra-red (NDIR) analyzer was used to measure CO₂ concentrations following the guidelines of U.S. EPA Method 3A, "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from a Stationary Source (Instrumental Analyzer Procedure)", in conjunction with Performance Specification No. 2 of Appendix B, 40 CFR 60. The analyzer was set at 25% instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C. Calibration error results are presented in Appendix A.

The CO_2 measurement principle utilizes a non-dispersive infrared (NDIR) method to measure the absorption of specific wavelengths of infrared radiation (IR) by the gas molecules through the use of the Gas Filter Correlation (GFC) technique. Radiation from an infrared source is chopped and then passed through a gas filter that alternates between a sample gas and N_2 due to the rotation of a filter wheel. The radiation then enters a multiple optical pass sample cell where absorption by the sample gas occurs. The IR radiation exits the sample cell and passes through a narrow band pass interference filter to fall on an IR detector. The sample gas filter produces a reference beam, which cannot be further affected by sample gas in the sample chamber. The N_2 side of the filter wheel is transparent to IR radiation and



therefore produces a beam, which can be absorbed by the sample gas. The chopped detector signal is modulated by the alternation between the twogas filters with amplitude proportional to the concentration of sample gas in the sample chamber. Other gases do not cause modulation of the detector signal since they absorb both the reference and measuring beams equally. Thus, the Gas Filter Correlation System responds solely to the desired sample gas molecules.

3.2.2 Nitrogen Oxides

The nitrogen oxides concentration was determined using U.S. EPA Reference Method 7E "Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure)". A Thermal Environmental Instruments chemiluminescence analyzer was set at 100 ppm instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C.

The analyzer converts the NO_x to NO and then measures the light emitted by the NO with ozone gas. The sample gas is drawn into the analyzer by a vacuum pump, which partially evacuates the reaction chamber. The sample flows through a NO₂ to NO converter for NO_x analysis or may pass through the converter for NO analysis. The sample then flows through a temperature controlled critical orifice into the partially evacuated reaction chamber. Ambient air is drawn into the analyzer as a source for the generation of ozone. The air flows through a desiccant cartridge for drying, then through an ozone generator converting some of the oxygen to ozone. The ozonated air then flows through a temperature controlled critical orifice into the reaction chamber. The sample gas and the ozonated air are mixed in the reaction chamber, where the following reaction takes place:

$$NO + O_3 \rightarrow NO_2 + O_2 + hv$$

The intensity of the chemiluminescence is proportional to the concentration of NO in the reaction chamber. The light emitted by this chemiluminescent



reaction shines through a window in the chamber onto a photomultiplier tube (PMT). A spinning chopper wheel between the reaction chamber and the PMT allows the dark PMT output to be compared electronically with the chemically generated chemiluminescent PMToutput. The signal is processed electronically for recording the NO concentration (or NO_x if the converter is used). A nitrogen dioxide to nitrogen oxide conversion efficiency test was performed on the NTH NO_x analyzer on May 10, 2004. The results of the NO_x converter test can be found in Appendix D.

3.2.3 Data Acquisition System

Information and data collected with the STRATA data acquisition system (DAS) was used for subsequent data reduction and reporting using a Microsoft Excel spreadsheet program. In addition to providing an instantaneous display of analyzer responses, the DAS compiles the analyzer data collected, and documents analyzer calibrations for download to a floppy disc. The DAS logs the concentration every 0.125 seconds and determines a one minute average.

4.0 RELATIVE ACCURACY TEST AUDIT PROCEDURES

The RATA was conducted to meet the requirements of 40 CFR Part 60, Appendix B, Performance Specification 2 and with the sampling and calibration procedures specified in 40 CFR 60, Appendix A, U.S. EPA Methods 3A, 6C, 7E, and 19. All of the RATA testing was conducted while the boiler was operating at more than 50% capacity. As specified in P.S. 2, Section 13.2, the relative accuracy of the CEMS can be calculated on the RM (average emissions are greater than 50% of the emission standard) or on the applicable emission standard (average emissions during test are less than 50% of the emission standard). The Relative Accuracy for this test report is calculated on the RM method.



4.1 Continuous Emissions Monitoring System (Cargill)

The extractive continuous emissions monitoring systems (CEMS) at the Cargill facility is in operation to monitor emissions from Boiler 10 and is installed to comply with Permit 184-01A. The CEM system includes a data acquisition system to provide data averaging. Graphs with averages for each applicable test period are contained in Appendix B.

4.2 REFERENCE MONITORING SYSTEM (NTH)

The stack gas was extracted from the stack continuously through a heated stainless steel probe. Measurements were conducted at a three-point traverse (approximately 16.7%, 50% and 83.3% of the sample stream diameter). Twelve 21-minute test runs were conducted, and the best nine runs were used to calculate the relative accuracy (RA). A diagram of the reference monitoring system is illustrated in Figure 2.

Data collected from the NO_X, and CO₂ analyzers were averaged for each test run. NO_X concentrations are reported in parts per million (ppm), converted to an emission rate and reported as (lbs/MMBtu), using equation 19-6 of U.S. EPA Method 19 of Appendix A, 40 CFR 60. CO₂ concentrations are reported in percent (%).

5.0 DATA REDUCTION AND CALCULATIONS

The exhaust gas flow rate and other exhaust gas characteristics calculations were performed by importing raw test data into standardized Microsoft Windows $Excel^{TM}$ worksheets. Once in the worksheet, average values were determined and the data was manipulated to return the desired information. The following calculations and procedures detail the determination of CO_2 , and NO_x emission rates.



The raw concentrations drawn from the stack effluent by the NTH CEMS analyzers was corrected for zero and upscale sampling system bias checks. The following formula was used to determine the corrected CO_2 and NO_x concentrations.

$$C_{gas} = \left(\overline{C} - C_o\right) \times \frac{C_{ma}}{C_m - C_o} \qquad Eq. 6C - I$$

Where:

C_{gas} = Effluent gas concentration, dry basis, ppm.

 \overline{C} = Average gas concentration indicated by gas analyzer, dry basis, ppm.

C_m = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.

 C_{ma} = Actual concentration of the upscale calibration gas, ppm.

After correcting the concentration values, Equation 19-6 found in U.S. EPA Method 19 was used to determine the emission rate in terms of pounds per million Btu heat input:

$$E = C_{\text{(stack)}} \times 1.194 \times 10^{-7} \times F_c \times \left(\frac{100}{\%\text{CO}_{2d}}\right)$$
 Eq. 19 - 6

Where:

E = Mass emission rate of pollutant, $lb/10^6$ Btu.

 $C_{(stack)}$ = Concentration in the stack, ppm by volume, dry basis.

 F_c = Fuel Factor for natural gas. (Table 19-2: 1,040 dscf/ 10^6 Btu)

 $%CO_{2d}$ = Measured CO_2 concentration, dry basis, percent.

 1.194×10^{-7} = Conversion factor, (ppm NO_x to lb/scf NO_y)



6.0 RESULTS

The best nine out of twelve runs were used to calculate relative accuracy. 40 CFR Part 60, Appendix B, Performance Specification No. 2 states that a maximum of three sets of the test results may be rejected, so long as the total number of test results used is greater than or equal to nine. The rejected test runs are highlighted in Table 1.

The RATA results for the NO_x analyzer indicates that the unit is within the allowable limits of 40 CFR, Part 60, Performance Specifications (PS) 2, which state that Relative Accuracy limits are 20 percent of the mean value of the Reference Method (RM) test data. The NO_x Relative Accuracy (RA) for Boiler 10 was 3.8 percent, stated as Lb/MMbtu based on the RM (Table 1). The following information is appended:

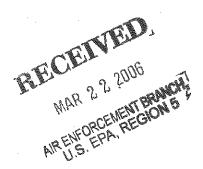
- A Boiler 10 Reference Monitor (NTH) RATA Test Data
- B Boiler 10 CEM System (Cargill) RATA Test Data
- C NTH Field Data
- D QA/QC data



TABLES
FIGURES
NOT CUPLED

TABLES





May 31, 2005

Mr. James Voss MDEQ – Air Quality Division SE Michigan District Office 38980 Seven Mile Road Livonia, Michigan 48152



Re: Compliance testing of EU BOILER 10 while firing natural gas at the Cargill Salt Inc. facility in St. Clair, Michigan

Dear Mr. James Voss;

Please find enclosed a copy of the test report for annual RATA testing. Cargill Salt Inc. retained NTH Consultants, Inc. to conduct the testing on EU Boiler 10. Testing was performed on May 24, 2005. The NOx Relative Accuracy (RA) for Boiler 10 was 17.7%.

If you have questions please contact me at 810-326-2763.

Sincerely:

Anthony Hoding EHS Manager

Enclosures

cc:

Ms. Karen Kajiya-Mills
Unit Supervisor – Compliance Support
Michigan Department of Environmental Quality
Air Quality Division
Constitution Hall – 3rd Floor, North
525 West Allegan Street

Don Chutas Wade Richards



Test Report for Compliance Relative Accuracy Testing Of EU BOILER 10 at Cargill Salt, Inc. Permit No. 184-01A

Test Date: May 24, 2005

Prepared For:

Cargill Salt Inc.
916 South Riverside Avenue
St. Clair, Michigan 48079

Project Number 16-040481-00

May 27, 2005

Prepared by: NTH Consultants, Ltd. Todd Wessel (616) 575-1022

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EXECUTIVE SUMMARY

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_x) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only. The testing was conducted with Boiler 10 operating at greater than fifty percent (50%) of the steam ouput capacity (150,000 pounds per hour faceplate rating). Please note that NTH's Data Acquisition System (DAS) timestamp was 5 minutes ahead of Cargill's DAS timestamp.

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B. The results of all testing can be found in Table 1, located at the end of this report, and is summarized below.

EXECUTIVE SUMMARY TABLE SUMMARY OF LB/MMBTU RELATIVE ACCURACY RESULTS

SOURCE	TEST PARAMETER	RELATIVE ACCURACY (based upon reference method)	ALLOWABLE LIMITS (based on mean value)
EU BOILER 10	NO _x	17.7 %	20 %



1.0 INTRODUCTION

NTH Consultants, Ltd. (NTH) was retained by Cargill Salt Inc. (Cargill) to conduct a Relative Accuracy Test Audit (RATA) of the Nitrogen Oxide (NO_x) and Diluent Continuous Emission Monitoring System (CEMS) at the Cargill facility located in St. Clair, Michigan. The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db. The boiler designated as EU-BOILER-10 (Boiler 10), is capable of firing natural gas (NG) or Number 2 Fuel Oil (serving as a backup fuel source). Boiler 10 was tested while firing NG only.

The testing was performed in accordance with specifications stipulated in 40 CFR, Part 60, Subpart Db, and Appendix A, U.S. EPA Reference Methods 3A, 7E, and 19. The RATA was performed in accordance with Performance Specification 2 stipulated in 40 CFR, Part 60, Appendix B.

NTH personnel Todd Wessel and Leona Taylor performed the compliance testing on May 24, 2005. The testing was coordinated through Mr. Wade Richards of Cargill.

2.0 PROCESS DESCRIPTION

The process boiler identified as EU BOILER 10 is a Nebraska N2S-7-89-ECON model that is designed to combust either NG or No. 2 fuel oil. The primary fuel used in the boiler is NG, with the No. 2 fuel oil serving as a backup fuel source (annual capacity factor for No. 2 oil firing is 10% or 1,176,526 gallons/year). The boiler is used to provide process steam for various plant operations and is rated at a maximum heat input of 184 Mmbtu/hour and a steam output capacity of 150,000 pounds per hour. When firing NG with an approximate heating value of 1,050 Btu/scf, the boiler will consume about 175,238 scf of NG per hour at full load.



3.0 EMISSION RATE SAMPLING AND ANALYTICAL PROCEDURES

The testing was performed to demonstrate compliance with the Michigan Department of Environmental Quality (MDEQ) Permit No. 184-01A, SRN: A6240 and the requirements of U.S. EPA Code of Federal Regulations (CFR) Part 60, Subpart Db.

The following U.S. EPA test methods were used during the emission rate testing:

- Method 3A Determination of Oxygen and Carbon Dioxide Concentrations in Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 6C Determination of Sulfur Dioxide Emissions From Stationary Sources (quality control requirements of 6C applied to Methods 3A and 7E);
- Method 7E Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure);
- Method 19 Determination of Sulfur Dioxide Removal Efficiency and Particulate
 Matter, Sulfur Dioxide, and Nitrogen Oxide Emission Rates

3.1 FLUE GAS COMPOSITION DRY MOLECULAR WEIGHT

The flue gas composition was determined using U.S. EPA Reference Method 3A "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)." The carbon dioxide was used for flue gas composition and pound per million british thermal unit (Lb/MMbtu) emission calculations.

3.2 CONTINUOUS EMISSION MONITORING SYSTEM (CEMS)

For all CEMS sampling, the NTH Reference Monitors (RM) require that the effluent gas sample be conditioned to eliminate any possible interference (i.e., water vapor and/or particulate matter) before being transported and injected into each analyzer. All components of the sampling system which contact the sample were constructed of Type 316 stainless steel or Teflon. The signal output from each monitor was connected to a computerized data acquisition system (DAS). The CO₂ and NO_x sample collection system consisted of a heated probe with a particulate filter, heated sample line, a



moisture removal trap, a secondary particulate filter and a sample pump. The sample was collected from the stack and routed through a distribution manifold for delivery to the analyzers. The configuration of the sampling system allowed for the injection of calibration gases directly to the analyzers or through the sampling system. All monitors in use were calibrated with U.S. EPA Protocol No. 1 calibration gases and operated to insure that zero drift, calibration gas drift, and calibration error met the specified method requirements. Response times were determined for each analyzer in use and found to be acceptable (30 seconds). Twelve test repetitions (each repetition 21 minutes in duration) were performed. A schematic of the CEMS is shown in Figure 2. Copies of all Protocol No. 1 gas standard certifications are in Appendix D.

3.2.1 Carbon Dioxide

A Horiba Model CMA 331A non-dispersive infra-red (NDIR) analyzer was used to measure CO₂ concentrations following the guidelines of U.S. EPA Method 3A, "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from a Stationary Source (Instrumental Analyzer Procedure)", in conjunction with Performance Specification No. 2 of Appendix B, 40 CFR 60. The analyzer was set at 25% instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C. Calibration error results are presented in Appendix A.

The CO₂ measurement principle utilizes a non-dispersive infrared (NDIR) method to measure the absorption of specific wavelengths of infrared radiation (IR) by the gas molecules through the use of the Gas Filter Correlation (GFC) technique. Radiation from an infrared source is chopped and then passed through a gas filter that alternates between a sample gas and N₂ due to the rotation of a filter wheel. The radiation then enters a multiple optical pass sample cell where absorption by the sample gas occurs. The IR radiation exits the sample cell and passes through a narrow band pass interference filter to fall on an IR detector. The sample gas filter produces a reference beam, which



cannot be further affected by sample gas in the sample chamber. The N_2 side of the filter wheel is transparent to IR radiation andtherefore produces a beam, which can be absorbed by the sample gas. The chopped detector signal is modulated by the alternation between the twogas filters with amplitude proportional to the concentration of sample gas in the sample chamber. Other gases do not cause modulation of the detector signal since they absorb both the reference and measuring beams equally. Thus, the Gas Filter Correlation System responds solely to the desired sample gas molecules.

3.2.2 Nitrogen Oxides

The nitrogen oxides concentration was determined using U.S. EPA Reference Method 7E "Determination of Nitrogen Oxides Emissions From Stationary Sources (Instrumental Analyzer Procedure)". A Thermal Environmental Instruments chemiluminescence analyzer was set at 100 ppm instrument span and calibrated before the RATA with zero nitrogen and high range span gas (80 to 100% of span). Following calibration, a mid range gas (40 to 60% of span) was introduced. The response error did not exceed 2% of the instrument span, as required by Method 6C.

The analyzer converts the NO_x to NO and then measures the light emitted by the NO with ozone gas. The sample gas is drawn into the analyzer by a vacuum pump, which partially evacuates the reaction chamber. The sample flows through a NO₂ to NO converter for NO_x analysis or may pass through the converter for NO analysis. The sample then flows through a temperature controlled critical orifice into the partially evacuated reaction chamber. Ambient air is drawn into the analyzer as a source for the generation of ozone. The air flows through a desiccant cartridge for drying, then through an ozone generator converting some of the oxygen to ozone. The ozonated air then flows through a temperature controlled critical orifice into the reaction chamber. The sample gas and the ozonated air are mixed in the reaction chamber, where the following reaction takes place:

$$NO + O_3 \rightarrow NO_2 + O_2 + hv$$



The intensity of the chemiluminescence is proportional to the concentration of NO in the reaction chamber. The light emitted by this chemiluminescent reaction shines through a window in the chamber onto a photomultiplier tube (PMT). A spinning chopper wheel between the reaction chamber and the PMT allows the dark PMT output to be compared electronically with the chemically generated chemiluminescent PMToutput. The signal is processed electronically for recording the NO concentration (or NO_x if the converter is used). A nitrogen dioxide to nitrogen oxide conversion efficiency test was performed on the NTH NO_x analyzer on May 23, 2004. The results of the NO_x converter test can be found in Appendix D.

3.2.3 Data Acquisition System

Information and data collected with the STRATA data acquisition system (DAS) was used for subsequent data reduction and reporting using a Microsoft Excel spreadsheet program. In addition to providing an instantaneous display of analyzer responses, the DAS compiles the analyzer data collected, and documents analyzer calibrations for download to a floppy disc. The DAS logs the concentration every 0.125 seconds and determines a one minute average.

4.0 RELATIVE ACCURACY TEST AUDIT PROCEDURES

The RATA was conducted to meet the requirements of 40 CFR Part 60, Appendix B, Performance Specification 2 and with the sampling and calibration procedures specified in 40 CFR 60, Appendix A, U.S. EPA Methods 3A, 6C, 7E, and 19. All of the RATA testing was conducted while the boiler was operating at more than 50% capacity. As specified in P.S. 2, Section 13.2, the relative accuracy of the CEMS can be calculated on the RM (average emissions are greater than 50% of the emission standard) or on the applicable emission standard (average emissions during test are less than 50% of the emission standard). The Relative Accuracy for this test report is calculated on the RM method.



4.1 Continuous Emissions Monitoring System (Cargill)

The extractive continuous emissions monitoring systems (CEMS) at the Cargill facility is in operation to monitor emissions from Boiler 10 and is installed to comply with Permit 184-01A. The CEM system includes a data acquisition system to provide data averaging. Graphs with averages for each applicable test period are contained in Appendix B.

4.2 REFERENCE MONITORING SYSTEM (NTH)

The stack gas was extracted from the stack continuously through a heated stainless steel probe. Measurements were conducted at a three-point traverse (approximately 16.7%, 50% and 83.3% of the sample stream diameter). Twelve 21-minute test runs were conducted, and the best nine runs were used to calculate the relative accuracy (RA). A diagram of the reference monitoring system is illustrated in Figure 2.

Data collected from the NO_X , and CO_2 analyzers were averaged for each test run. NO_X concentrations are reported in parts per million (ppm), converted to an emission rate and reported as (lbs/MMBtu), using equation 19-6 of U.S. EPA Method 19 of Appendix A, 40 CFR 60. CO_2 concentrations are reported in percent (%).

5.0 DATA REDUCTION AND CALCULATIONS

The exhaust gas flow rate and other exhaust gas characteristics calculations were performed by importing raw test data into standardized Microsoft Windows ExcelTM worksheets. Once in the worksheet, average values were determined and the data was manipulated to return the desired information. The following calculations and procedures detail the determination of CO₂, and NO_x emission rates.



The raw concentrations drawn from the stack effluent by the NTH CEMS analyzers was corrected for zero and upscale sampling system bias checks. The following formula was used to determine the corrected CO_2 and NO_x concentrations.

$$C_{gas} = \left(\overline{C} - C_o\right) \times \frac{C_{ma}}{C_m - C_o} \qquad Eq. 6C - 1$$

Where:

 C_{ma}

 C_{eas} = Effluent gas concentration, dry basis, ppm.

 \overline{C} = Average gas concentration indicated by gas analyzer, dry basis, ppm.

C_m = Average of initial and final system calibration bias check responses for the upscale calibration gas, ppm.

Actual concentration of the upscale calibration gas, ppm.

After correcting the concentration values, Equation 19-6 found in U.S. EPA Method 19 was used to determine the emission rate in terms of pounds per million Btu heat input:

$$E = C_{\text{(stack)}} \times 1.194 \times 10^{-7} \times F_c \times \left(\frac{100}{\% \text{CO}_{2d}}\right)$$
 Eq.19-6

Where:

E = Mass emission rate of pollutant, $lb/10^6$ Btu.

 $C_{(stack)}$ = Concentration in the stack, ppm by volume, dry basis.

 F_c = Fuel Factor for natural gas. (Table 19-2: 1,040 dscf/10⁶Btu)

 $%CO_{2d}$ = Measured CO_2 concentration, dry basis, percent.

 1.194×10^{-7} = Conversion factor, (ppm NO_x to lb/scf NO_x)



6.0 RESULTS

The best nine out of twelve runs were used to calculate relative accuracy. 40 CFR Part 60, Appendix B, Performance Specification No. 2 states that a maximum of three sets of the test results may be rejected, so long as the total number of test results used is greater than or equal to nine. The rejected test runs are highlighted in Table 1.

The RATA results for the NO_x analyzer indicates that the unit is within the allowable limits of 40 CFR, Part 60, Performance Specifications (PS) 2, which state that Relative Accuracy limits are 20 percent of the mean value of the Reference Method (RM) test data. The NO_x Relative Accuracy (RA) for Boiler 10 was 17.7 percent, stated as Lb/MMbtu based on the RM (Table 1).

The following information is appended:

- A Boiler 10 Reference Monitor (NTH) RATA Test Data
- B Boiler 10 CEM System (Cargill) RATA Test Data
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TABLE